भारतीय मानक

Indian Standard

IS 9136 : 2022

कैल्शियम कॉम्प्लेक्स ग्रीस — विशिष्टि

(*दूसरा पुनरीक्षण*)

Calcium Complex Grease — Specification

(*Second Revision*)

ICS 75.100

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110 002
www.bis.gov.in www.standardsbis.in

July 2022 **Price Group 7**

Lubricants and their Related Products Sectional Committee, PCD 25

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Lubricants and their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1978 in view of the fact that, while preparing the lithium soap grease specification, it was felt that lithium hydroxide being imported material, there could be possible difficulties in its procurement. In the first revision in 1993, various requirements for the base oil (mineral) were incorporated in order to reflect the improved quality of base oil to be used for the purpose of manufacturing the product. Further, various requirements given were modified for better appreciation of the product in its application.

In this second revision, the requirements of viscosity index, flash point, water washout test and Emcor rust test have been upgraded for all the grades.

The composition of the committee responsible for the formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CALCIUM COMPLEX GREASE — SPECIFICATION

(*Second Revision*)

1 SCOPE

This standard prescribes the requirements and the method of sampling and tests for calcium complex grease suitable for automotive and industrial purposes.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 1447 (Part 3) : 2021	Methods of sampling of petroleum and its products: Part 3 Method of sampling of semi-solid and solid petroleum products (*second revision*)
1448	Methods of test for petroleum and its products
(Part 25/Sec1) : 2018/ISO 3104 : 1994	Transparent and opaque liquids Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
(Part 51) : 1963	Copper strip corrosion test for lubricating greases
(Part 52) : 2017/ISO 2176 : 1995	Drop point (*second revision*)
(Part 53) : 1979	Determination of acidity and alkalinity of greases (*first revision*)
(Part 56) : 2013/ISO 2909 : 2002	Calculation of viscosity index from kinematic viscosity (*third revision*)
(Part 59) : 1991	Determination of mineral oil content in greases (*second revision*)
(Part 60) : 1994	Consistency of lubricating greases by cone penetrometer (*second revision*)
(Part 69) : 2019/ISO 2592 : 2017	Determination of flash and fire points — Cleveland open cup method (*second revision*)
(Part 89) : 1979	Test for thermal stability of lubricating greases
(Part 90) : 2008/ISO 11009 : 2000	Petroleum products and lubricants — Determination of water washout characteristics of lubricating greases (*first revision*)
(Part 94) : 2019	Test for oxidation stability of lubricating grease by oxygen pressure vessel method (*first revision*)
(Part 164) : 2018	Determination of the leakage tendencies of automotive wheel bearing grease under accelerated conditions
(Part 170) : 2018/ISO	Determination of the extreme-pressure and anti-

IS No.	*Title*
20623 : 2003	wear properties of fluids — Four ball method (European conditions)

3 GRADES

The material shall be of the following three grades:

a) Grade 1,

b) Grade 2, and

c) Grade 3.

4 REQUIREMENTS

4.1 General

The material shall be a smooth and homogeneous preparation free from objectionable odour and visible impurities. It shall be free from resins, rosin oil, rosinate, tar oil and fillers such as clay, asbestos, talc and from products gelled with bentonite and silica gel.

4.2 Composition

The material shall be prepared from refined mineral lubricating oil and a suitable calcium soap-salt gelling agent.

4.2.1 The mineral oil, extracted from the grease by method A prescribed in IS 1448 (Part 59), shall comply with the requirements given in Table 1.

Table 1Requirements of Mineral Oil

(*Clause* 4.2.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Part of IS 1448
(1)	(2)	(3)	(4)
i)	Kinematic viscosity at 40 ºC, mm^2/s	135-165	Part 25/Sec 1
ii)	Viscosity index, *Min*	90	Part 56
iii)	Flash point, ºC, *Min* (COC)	180	Part 69

NOTE — 1 cSt = 1 mm^2/s.

4.3 The material shall also comply with the requirements given in Table 2 when tested according to the methods given in Parts of IS 1448, given in co1 6 of the table.

4.4 Keeping Quality

The keeping quality of the material shall be such that when stored in original sealed containers under normal conditions, it shall retain the properties given in the specification for not less than one year from the date of manufacture.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in metal or any other suitable containers as agreed to between the purchaser and the supplier.

5.2 Marking

Material shall be marked with the following information:

a) Name and type of material;

b) Manufacturer's name, initials or trade-mark, if any;

c) Net mass of material;

d) Identification in code or otherwise to enable the lot of consignment or manufacture to be traced back from records; and

e) Any other statutory requirements.

5.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 3).

Table 2 Requirements for Calcium Complex Grease

(*Clause* 4.3)

Sl No.	Characteristic	Requirement for			Methods of Test, Ref to 'Part' of IS 1448 /ASTM/IP/Annex
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Consistency of worked grease at 25 ± 0.5 ºC				
	a) 60 strokes	355-385	310-340	265-295	Part 60
	b) 100 000 strokes	shall not differ by more than 30 units from 60 stroke value			
ii)	Alkalinity [as $Ca(OH)_2$][1)] percent by mass, *Max*	0.5	0.5	0.5	Part 53
iii)	Free organic acidity (as oleic)[1)], percent by mass, *Max*	0.5	0.5	0.5	Part 53
iv)	Copper strip corrosion for 24 h at 100 ºC	← Negative →			Part 51
v)	Drop point, °C, *Min*	260	260	260	Part 52
vi)	Oxidation stability (100 h) at 100 ºC, drop in pressure, kg/cm^2, *Max*	0.7	0.7	0.7	Part 94
vii)	Leakage and deposit forming tendencies (wheel bearing test)				Annex A
	a) Leakage by mass, g, *Max*	NA	5.0	5.0	
	b) Deposit in wheel bearing races or rollers	← shall be free from deposits →			
	c) Evidence of abnormal change in consistency or structure of material	Not limited, but observations are to be reported			

Sl No.	Characteristic	Requirement for			Methods of Test, Ref to 'Part' of IS 1448 /ASTM/IP/Annex
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
	d) Indication of dry running of races	Not limited, but observations are to be reported			
viii)	Resistance to water wash out at 80 ºC, percent loss in mass, *Max*	NA	5.0	5.0	Part 90
ix)	Heat stability, percent loss in mass, *Max*	NA	5.0	5.0	Part 89
x)	Freedom from deleterious particles, permitted number of scratches, *Max*	10	10	10	D1404
xi)	Timken OK load, kg, *Min*	18	18	18	D2509
xii)	Four ball weld load, kg, *Min*	200	200	200	Part 170
xiii)	Emcor rust test rating, *Max*	0.0	0.0	0.0	IP 220
xiv)	Low temperature torque test at -18 ºC, starting torque/Running torque	Starting and running torque to be reported			IP 186

1) If additives such as heavy metal soaps, rust inhibitors etc, are present acidity/alkalinity values need not to be reported.

ANNEX A

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF THE LEAKAGE TENDENCIES OF AUTOMOTIVE WHEEL BEARING GREASES

B-1 GENERAL

This test method describes the assessment of the leakage tendencies of wheel bearing greases when tested under prescribed laboratory conditions.

B-2 TERMINOLOGY

B-2.1 Lubricating Grease

A semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant.

B-2.1.1 A two-phase system is formed by the dispersion of thickener, immobilizing the liquid lubricant, due to surface tension and other physical forces. Other ingredients are added to get other special properties.

B-2.2 Lubricant

Any material, which, when interposed in between two surfaces, lowers down the friction or wear between them.

B-2.3 Thickener

In lubricating grease, a substance used to form the structure of product, composed of finely-divided particles dispersed in a liquid lubricant.

B-2.3.1 The solid thickener can be spheres (such as certain non-soap thickeners) or fibers (such as various metallic soaps) or plates. The solid particles are insoluble or, at the most, only very slightly soluble in the liquid lubricant. Solid particles should generally be uniformly dispersed, extremely small, and capable of forming a gel-like, relatively stable structure with the liquid lubricant.

B-2.4 Automotive Wheel Bearing Grease

A lubricating grease categorically formed to lubricate automotive wheel bearings at relatively high temperature of grease and bearing speed.

B-2.5 Leakage of Wheel Bearing Grease

Segregation and overflow of oil or grease from the bulk grease charge, activated by high temperature and bearing rotation.

B-3 PRINCIPLE

B-3.1 The grease is distributed in a modified front-wheel hub and spindle assembly. Spindle temperature is raised to and maintained at 105 $\pm$ 1.2 °C. The hub is rotated at a speed of 660 $\pm$ 30 rpm for 6 h $\pm$ 5 min. Leakage of oil or grease, or both, is measured, and at the end of the test, the condition of the bearing surface is noted.

B-3.2 A screening device is provided by the test method that allows differentiation among products of distinctly different leakage characteristics. This test method is not equivalent to long time service tests and it is also not aimed at distinguishing between wheel bearing greases showing similar or border line leakage.

NOTE — Skilled operators may observe significant changes in other important grease characteristics that occur during the test. Such additional information can be of special interest to individual operators. The observations, however, cannot be

used effectively for quantitative rating, as these are subject to differences in personal judgment among operators.

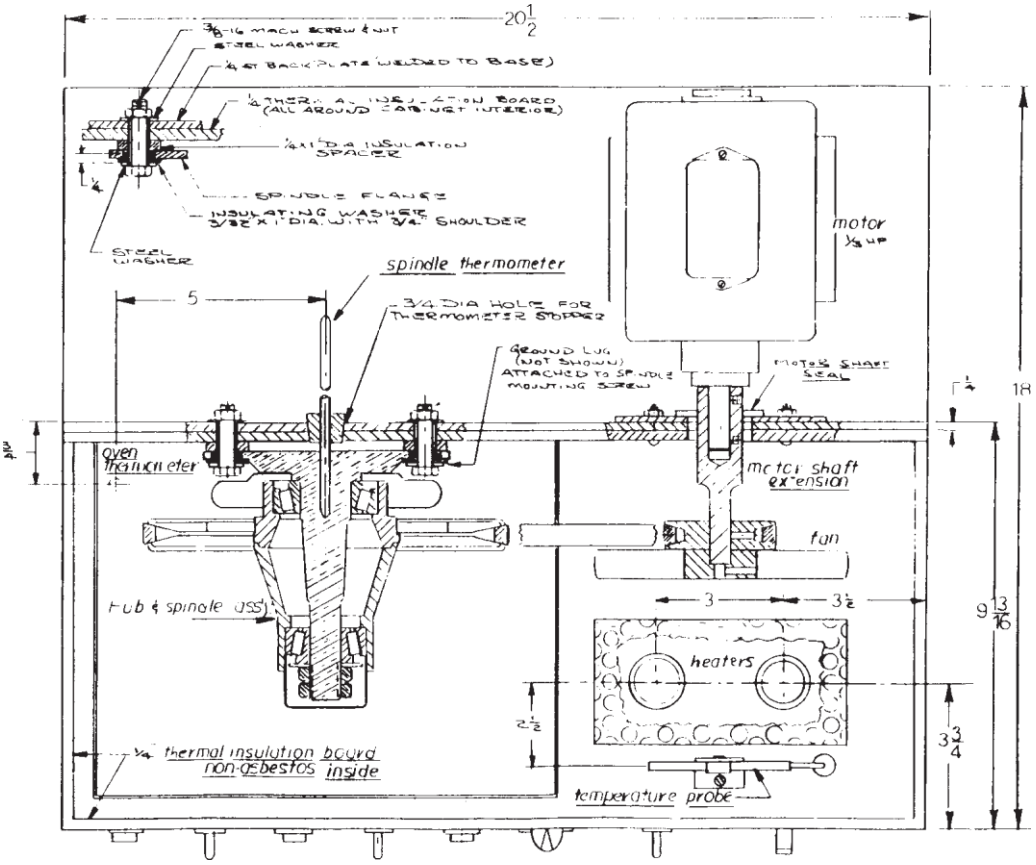
B-4 APPARATUS

B-4.1 The suitable apparatus is shown in Fig. 1 and Fig. 2. The tester has a special front wheel hub and spindle assembly and the hub is rotated by an electric motor using a V-belt drive. The assembly is encased in a thermostatically controlled air bath. There are means to measure both ambient (cabinet) and spindle temperatures. A torque wrench is also required, which is suitable for use on 31.75 mm hexagonal nuts.

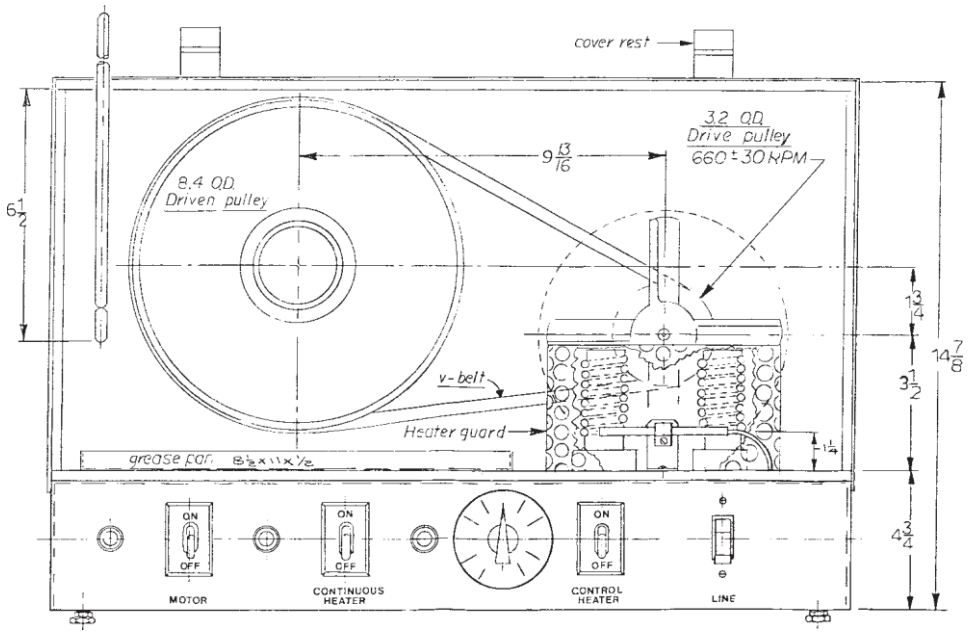
B-4.2 The apparatus (spindle, case, and motor) must be electrically grounded, otherwise the thermocouples will not function due to accumulated static charges. Provision is made for this, as shown in Fig. 2.

B-4.3 Usually machine having 660 W heaters will provide sufficient heat input to attain the temperatures in the specified time intervals. However, if proper balance cannot be obtained, heaters with required wattage can be substituted.

FIG. 1 APPARATUS FOR TESTING LEAKAGE TENDENCIES OF WHEEL BEARING GREASES



All dimensions in inch



All dimensions in inch

FIG. 2 DETAILS OF MAIN ASSEMBLY

B-4.4 Main Assembly

The main assembly shall consist of spindle assembly mounted in a thermostatically controlled air bath and a special front-wheel hub. The assembly is arranged so that the hub will be rotated by an electric motor through a V-belt drive, as shown in Fig. 2. One continuous and one intermittent heater shall be mounted on the base of the apparatus and shall be controlled thermostatically.

B-4.5 Bearing Spindle

The spindle shall be constructed as shown in Fig. 3.

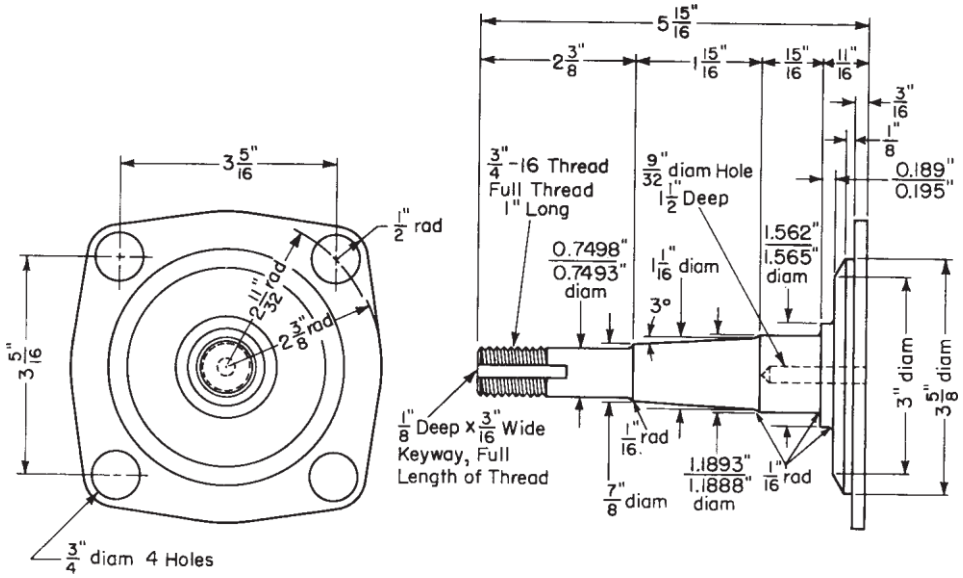


FIG. 3 DETAILS OF MAIN SPINDLE

B-4.6 Bearing Hub

The bearing hub shall be constructed as shown in Fig.4.

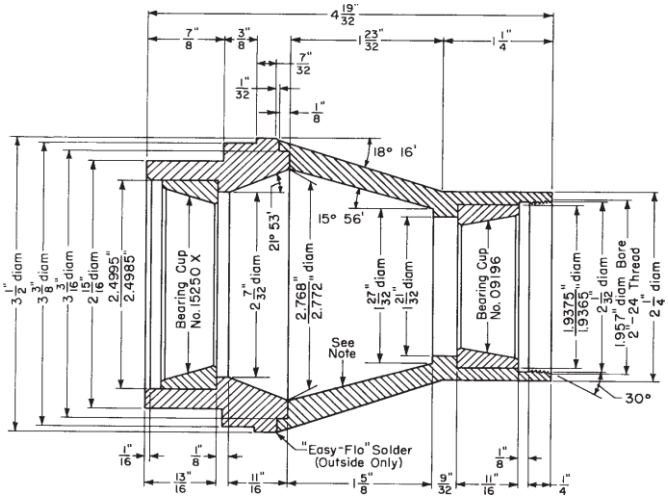


FIG. 4 DETAILS OF BEARING HUB

B-4.7 Leakage Collector

A cup-shaped ring shall be used as a leakage collector to catch any leaked grease from the inner end of the hub, as shown in Fig. 5. The ring shall be removable providing a method for determining the grease loss. The ring is held in place by the large bearing.

NOTE — The regular grease retainer is not suitable for this purpose, as it is required to accelerate any leakage that may occur. Also retainers are frequently found to be defective.

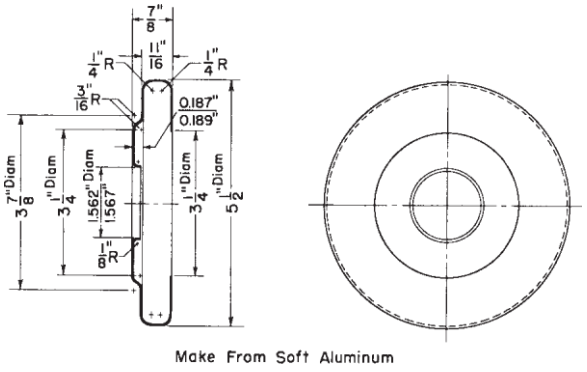


FIG. 5 DETAILS OF GREASE COLLECTOR

B-4.8 Fan

The fan shall be constructed as shown in Fig. 6.

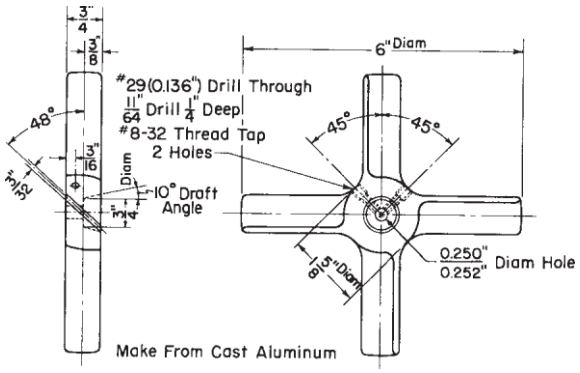


FIG. 6 DETAILS OF FAN

NOTE — Polish inside tapered surfaces with waterproof paper. Do not round off sharp corners when polishing. Make both parts of hub assembly from 8.89 cm round cold drawn steel rod.

B-4.9 Test Bearings

B-4.9.1 *Inner Bearing* (*Tapered Roller*) — Timken 15118. The corresponding cup No. 15250.

B-4.9.2 *Smaller Outer Bearing* — Timken 09074. The corresponding cup No. 09196.

B-5 REAGENT

B-5.1 Heptane — 99.87 percent purity.

WARNING — Flammable. Harmful if inhaled.

B-6 PROCEDURE

B-6.1 Weigh 90 ± 1 g of sample on a flat plate. Pack 2 ± 0.1 g of grease in the small bearing using a spatula. Similarly, pack 3 ± 0.1 g of grease in the large bearing.

B-6.2 On the inside of the hub, distribute the balance of the test grease (85 g) in a uniform layer. Apply a thin film of grease to the bearing races in the hub.

NOTES

1 A narrow, wedge-cut spatula is suitable for packing the bearings.

2 Rest of the grease will fill the hub sufficiently, even with the races and except for very fibrous greases, it can be distributed quickly and uniformly with a spatula of 150 mm blade.

B-6.3 Weigh the leakage collector and the hub cap to the nearest 0.1 g separately. Put the large (inner) bearing and leakage collector in the proper position on the spindle. Put small (outer) bearing and the hub and on the spindle, followed by the loose-fitting retainer ring. Tighten the hexagonal nut which holds the hub assembly in place, applying a torque of 6.8 ± 0.1 N·m using the torque wrench. Then back off the hexagonal nut 60 ± 5° (or one flat), and lock it in position with a second hexagonal nut. Put screws on the hub cap and V-belt on the pulleys, and close the cabinet.

Caution — Inspect all the grease collectors carefully to make sure that the inner lip is flush with the sealing face. Otherwise, this lip will interfere with the correct seating of the inner bearing.

Caution — Prevent contact between grease pack and spindle, while assembling the packed hub on the spindle.

Caution — From time to time, the drive pulley and the driven pulley should be checked for alignment. Misalignment can introduce leakage variations.

NOTE — Excessive end play of the hub assembly is sometimes due to worn bearings. Therefore new bearings, both cups and cones, should be installed after each 250 tests, or sooner if inspection indicates wear or other damage to the bearings.

B-6.4 Close the cabinet and turn on both the heaters and motor. Operate at a speed of 660 ± 30 rpm for 6 h ± 5 min. The spindle temperature to be raised to and then maintained at 105 ± 1.4 °C during the test period. Maintain the ambient temperature at 115 ± 3 °C to obtain the spindle temperature of 105 ± 1.4 °C. Keep the auxiliary heater on till an ambient or oven temperature of 115°C is attained. Keep the thermo regulator previously adjusted to maintain oven temperature of 115 °C, or to have a reproducible setting for this temperature. The ambient temperature of 115 ± 3 °C shall be attained within 15 ± 5 min. The spindle temperature of 105 ± 1.4 °C shall be attained within 60 ± 10 min. These two combining result in the spindle maintaining at 105 ± 1.4 °C for 5 h ± 15 min.

Caution — Rate of heating can be affected by drafts. Therefore, location of the tester should be carefully chosen.

B-6.5 Shut off the power after 6 h from the time motor and heater are turned on and dismantle the apparatus while hot. Wear appropriate protective clothing while handling the hot equipment.

B-6.6 Let the apparatus cool and measure the weight of the hubcap and leakage collector separately to the nearest 0.1 g.

NOTE — In case of overflow from leakage collector, the amount of overflow grease or oil, or both, should be weighed and included in the reported total leakage.

B-6.7 Wash the two bearings in heptane, at room temperature for at least 2 min, to

remove the grease. Inspect for varnish, gum, or lacquer-like deposits.

NOTE — In case of some wheel bearing greases, it will be found that the soaps are not completely washed from the bearings with *n*-heptane and film of soap may remain on the bearings. However, such film can be easily distinguished from varnish, gum, or lacquer-like deposits resulting from deterioration of the lubricant.

B-7 REPORT

B-7.1 Report the total amount of leakage of grease or oil, or both, into the collector and into the hubcap. Report the presence of any adherent deposit of varnish, gum, or lacquer-like material on the bearing surface, which is evident after removal of the grease.

B-8 PRECISION AND BIAS

B-8.1 Repeatability

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Leakage in Area of	*Acceptable Difference*
2 g	1.5 g
15 to 20 g	9 g

B-8.2 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

Leakage in Area of	*Acceptable Difference*
2 g	4 g
15 to 20 g	9 g

B-8.3 Bias

The procedure in this test method for measuring leakage tendencies of automotive wheel bearing greases has no bias because the value of leakage can be defined only in terms of a test method.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Lubricants and their Related Products Sectional Committee, PCD 25

Organization	*Representative(s)*
Gulf Oil Lubricants India Limited, Mumbai	DR Y. P. RAO (***Chairman***)
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Member Secretary
SHRI ARIDAMAN
Scientist 'B', PCD, BIS

Automotive and Industrial Greases Subcommittee, PCD 25 : 3

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Association of State Road Transport Undertakings, New Delhi	REPRESENTATIVE
Bajaj Auto Limited, Pune	REPRESENTATIVE
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Bureau of Indian Standards

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Review of Indian Standards

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
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